

A monitored Geiger-counter X-ray powder diffractometer with automatic recording

The publication by Bond⁽¹⁾ and Benedict⁽²⁾ of an account of their single-crystal automatic X-ray diffractometer prompts us to report another automatic instrument designed for highly accurate recording of powder diffraction patterns. "Step-scanning" of each diffraction line now becomes necessary, and, in common with the single-crystal instrument, techniques for rapid movement between lines and for "retracking" are used. A description of our equipment follows.

The apparatus will record a pattern on a chart in the usual way, but accurate determinations of the peak position and intensity of each diffraction line, as well as its centre of gravity and its integrated intensity, require more precise information than the chart can yield. Step-scanning is employed for this accurate work, and a direct plot of the profile of each line may be made from the record of such a scan if Geiger-counter dead-time losses are eliminated by incorporating the monitoring circuit of Eastabrook and Hughes.⁽³⁾ This circuit also corrects the record for variations in the primary beam intensity, and reduces to about one-third the cosmic background recorded.

The fundamental scanning interval is 0.02° (2θ) and Dekatron counting tubes register the diffracted beam intensity. The recording is made by photographing the faces of these tubes, together with a register of the angle 2θ , at the end of each step. As in the apparatus of Bond and Benedict, the counter arm is arranged to move rapidly over the background region, and to traverse a line more slowly, but the mechanism of this process is rather different in our arrangement. The background is "stepped" without monitoring or recording in 0.2° steps; the counter remains stationary in each position only long enough to enable a counting-rate meter to reach a steady state. If the rate in a particular position is below a preset level, a further step of 0.2° is taken. If not, an electronic circuit operates a relay which reverses the stepping process so that twenty steps of 0.02° are taken without pause to bring the counter to the position before that in which the background was last sampled. A monitored 0.02° step-scan, with photographic registration, is then initiated and continues through the line until the counting rate falls below the preset level; a further ten steps of 0.02° are similarly recorded and then the background stepping recommences. From the photographic record of this process the line profile and the immediate background on either side may be plotted. The total number of counts made during the entire 0.02° step-scan is separately recorded on the film to assist in the rapid evaluation of the integrated intensity of the line. The whole process of scanning a diffraction pattern is automatic, and is controlled by a sequential circuit which uses Post Office relays. Facilities for continuous step-scanning as well as manual recording are also available.

The camera, which is of special design, can store 200 ft of 35 mm film which is led under a shutter into a cassette. When the camera is placed in the main equipment rack, the "leader" of film is automatically wound on, and the camera is locked in the rack. When a record is completed, the length of film between the shutter and the cassette, which carries the latter part of the record, is automatically wound into the cassette before the camera can be withdrawn for removal of the cassette and processing of the record. Devices for indicating the number of exposures made, and the length of film remaining in the store, are also incorporated; the entire recording process ceases if the store of film becomes exhausted.

The apparatus has been developed with the aid of a grant from the crystallographic data fund held in trust by The Institute of Physics. It will be on show at The Physical Society's exhibition this year, and a full description of it will be published in due course.

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Calibration of a stroboscopic light.

In a previous issue of this *Journal* Lemlich* describes a method of calibrating a stroboscopic light over its entire range by utilizing a piece of machinery rotating at a single constant speed. It may be of interest to point out that tuning forks also provide a simple and accurate means of calibrating stroboscopic lights. Sets of tuning forks are not ordinarily available in machine shops, but are standard equipment in physical and physicochemical laboratories. They are regularly employed in this laboratory for the calibration of stroboscopic lights, which are in turn used for measuring the rotation speeds of air-driven and electrically driven centrifuges.

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